

Preparation of Magnetic Activated Carbon

Zhang Shirun (张世润) Liu Shouxin (刘守新) Fang Guizhen (方桂珍) Zhu Ying (朱莹)

Northeast Forestry University, Harbin 150040, P. R. China

Cheng Zhifen (程志芬)

Harbin Institute of Technology

Abstract Based on the special molecular structure of surfactant, paramagnetic material used as magnetic nucleus was coated with two layers of surfactant including an inner layer and an outer layer. Magnetic reagent can combine with activated carbon due to adsorption of hydrophilic radical of the outer surfactant layer. So activated carbon has magnetism. The effects of magnetic reagent dosage, pH and activator upon the magnetic susceptibility of activated carbon were studied. Concerning the optimal conditions, MR-20 is the best magnetic reagent. Concentration of CaCl_2 is 0.02M and pH 1.8-2.4. The physical properties of magnetic activated carbon vary with the types, dosage and magnetizing condition of magnetic reagent.

Key words: Activated Carbon, Magnetic Nucleus, Surfactant, Magnetic Reagent, Magnetic Activated Carbon.

Introduction

As one of an important industrial material, activated carbon has a marked capacity, giant specific surface, developed pore structure and stable chemical properties. It has played more and more important role in metallurgy, medicine, food and environment field. But there still have many problems in using activated carbon, such as separation. As usual filtration is adopt. But this method bring problem either blocking or losing. Compare to filtration, the use of magnetic separation was time saving, cheap, reduce hazards and improve quality, so people try using magnetic separation instead filtration. But it's impossible because of the low magnetic susceptibility of activated carbon. So, the preparation of magnetic activated carbon became an interesting subject of the reachers and users throughout the world. China, Japan and USA have done a lot of work on it, but all didn't get satisfied results. 1990, Tangshan Jianxin Activated Carbon Company used adhesive to make magnetic particle adhere to the surface of activated carbon. But because of the adhesive, the capacity of activated carbon decreased greatly. 1991, Wang Chonglin^[1], A researcher of Chinese Academy of Science Metal Institute prepared magnetic activated carbon by immersing activated carbon in a salt solution of Fe, Co, Ni, then immersing the C in a $(\text{NH}_4)_2\text{C}_2\text{O}_4$ solution, as last magnetizing the activated carbon in H_2 and N_2 at 600-1200 °C. Because this method need high temperature and pressure, it cost expensive. In addition to this, the magnetic activated carbon's total iron is too much higher and capacity decreased greatly. In 1992, Eto,

Takeo^[2] obtained magnetic activated carbon by dispersing activated carbon and magnetic material in a solution containing latex, CMC and water and coating the dispersion on activated carbon. Between 1988-1992, America researchers did a lot on this field, but got little^[3].

In this paper, based on the magnetic reagent technology, produced magnetic activated carbon which still had high capacity and giant surface area.

Experiment

Synthetic Principle of Magnetic Reagent

Magnetic reagent is a composite of magnetic materials included magnetite, ferrite, hematite, maghemite, pyrrhotite and metals, alloys and compounds containing iron, nickel or cobalt^[2] and surfactant coating with an inner layer and an outer layer. The functional groups of surfactant can react between magnetic material and other materials. Under suitable conditions, surfactant can serve as a bridge. Thus, the composite of magnetic materials and surfactants with the capabilities to couple other materials is magnetic reagent.

Fig. 1 is an ideal representation of magnetic reagent in water^[4].

The inner layer covered the particle is a monomolecular layer of a first water soluble, organic, heteropolar surfactant coating at least 3 carbon atoms and having a functional group which forms a bond with the magnetic particle on one end and a hydrophobic end. The outer layer covered the inner layer is a monomolecular of a second water soluble, organic, heteropo-

lar surfactant containing at least 3 carbon atoms and having a hydrophobic end bonded to the hydrophobic of the first surfactant and a functional group oriented outwardly toward water. Nonmagnetic particles were magnetized by contacting the surface with magnetizing reagent in an aqueous medium and adsorbing the surface with covered two layers surfactant magnetic particles. The first and second surfactants can be nonionic, anionic or cationic.

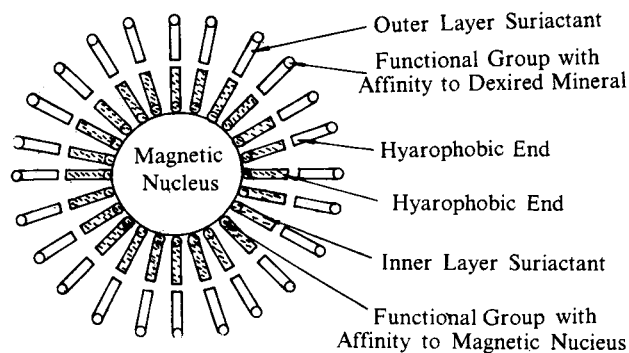
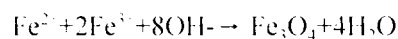


Fig. 1. The structure of magnetic reagent

Magnetic materials include magnetite, ferrite, hematite, maghemite, phrrhotite and metals, alloys and compounds containing iron, nickle or cobalt. The magnetic materials can be derived from various sources. For example, magnetite may be obtained from ores and prepared by grinding^[6,7,8] or by so called wet method. Colloidal magnetite can be precipitated by reacting solutions of ferrous and ferric salts with alkali metals^[9,10].



Synthetic Example of Magnetic Reagent

Magnetic particles were prepared by reacting between salt of ferrous and ferric and alkali. 3.6g $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$, 4.8g $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ were dissolved in 10 ml water and 10 ml of ammonia solution (56%) was added with rapid mixing to yield magnetite precipitates. The solution was heated to boiling. The magnetite particles were

collected on a magnet. Washed with water to remove the residual salts and a yield of about 2.7g of magnetic particles was obtained.

0.6g sodium lauryl sulfate was added to an aqueous slurry of the colloidal magnetic particles (2.7g magnetic particles in 10 ml water) and 5 drops of 5N sodium hydroxide were added. The slurry was stirred and heated at 92 °C for 50 min to aid the adsorption of lauric acid onto the magnetic particles. 1N hydrochloric acid was then added until pH reach 7.0. The agglomerates were concentrated held by a magnet and the solution decanted. The agglomerates were further washed with water to remove excess surfactant. 0.5g sodium disopropul dithiophosphates and sufficient water to make a total vollume of 10ml were added to the agglomerate to obtain a peptized product which the object product-magnetic reagent.

Preparation of Magnetic Activated Carbon

Washed activated carbon twice and then immersed in 0.02M CaCl_2 solution for 2 hours. Then adjust the pH of the solution to a suitable range, added the magnetic reagent (a certain amount magnetic reagent in water). Maintained at 60 °C for 2 hours. Washed activated carbon with water to remove excess of CaCl_2 and magnetic reagent. At last dry activated carbon at 104 °C for 2 hours. We'll get magnetic activated carbon.

Results and Discussion

Effect of Magnetic Reagent Kind

The same magnetic reagent had a different magnetic capability for different materials. So before preparation of magnetic activated carbon, we should determine optimal magnetic reagent.

Table 1 showed MR-20 was optimal magnetic reagent for preparation of activated carbon.

Table 1. Magnetic Capability for Activated Carbon of Various Magnetic Reagent

MS	MR								
	MR-3	MR-6	MR-9	MR-14	MR-15	MR-17	MR-19	MR-18	MR-20
pH									
2.0	9.705	38.431	41.203	50.061	48.700	15.384	24.868	4.680	72.136
5.5	5.178	17.115	37.945	24.865	46.531	15.390	19.755	1.258	44.850
7.2	4.794	17.005	36.744	21.245	32.624	11.478	9.092	4.016	38.764
10.7	6.524	17.045	18.911	6.691	24.488	6.102	9.314	3.501	38.689

MS = Magnetic susceptibility $\cdot 10^{10} \text{ emu/g}$

Effect of pH

When material particles had zero charge the pH was called the isoelectric point. When the pH is above the

isoelectric point, these particles carry negative charge and will adsorb or couple with a magnetic reagent having a cationic outer layer surfactant. When the pH below the isoelectric point, these particles carry a positive

change and will adsorb or couple with a magnetizing reagent having an anionic outer layer surfactant^[11].

Fig. 2 showed, when MR-20 was used for preparation of magnetic activated carbon, optimal pH range was 1.8-2.4.

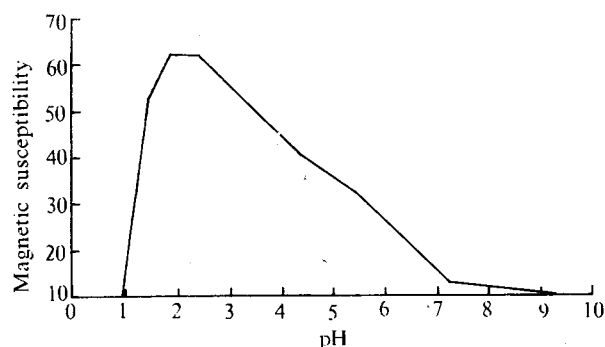


Fig. 2. The adsorption of MR-20 on activated carbon at various pH

Effect of Activator

The coupling between magnetic reagent and activated carbon was determined by these following factors^[12].

1) Non-polar Van de Waals for a relative weak attraction force which increase with the molecular weight of the outer layer surfactant.

2) Hydrogen bonding force.

3) Electrostatic bonding force.

4) Covalent bonding force.

The last three forces were quite strong, so we can add some salts such as CaCl_2 which can activate the adsorption through modifying the surface properties of activated carbon and were usually called activators. Fig. 3 show that magnetic enhancement can indeed be achieved with this approach.

Effect of Magnetic Reagent Dosage

It was shown in Fig. 4, in the range of study, the amount of adsorption was almost linearly proportional.

Main Indexes of Magnetic Activated Carbon

Table 2, 3, 4 showed, the main indexes of magnetic activated carbon were changed with the magnetic susceptibility change, but the adsorbing capacity changed just a little.

Conclusion

1. Used GH-16A, JX-01, GH-14 as raw materials to prepare magnetic activated carbon, MR-20 is the optimal magnetic reagent.

2. In this study, the optimal conditions for the preparation of magnetic activated carbon were: pH 1.8-2.4,

temperature 60 °C, CaCl_2 0.05M.

3. The kind of magnetic reagent, dosage, pH and activator had a little effect on the main indexes of magnetic activated carbon.

4. The magnetic material of magnetic activated carbon prepared by this method spread evenly. When grind under 200 mesh, the activated particle still be magnetic.

5. This kind of magnetic belong to soft magnetic material and it was absorbed only by magnet.

6. The success of this study provides a method for other nonmagnetic materials magnetized.

Table 2. Main Indexes of MGH-16A

Analytic index	1	2	3	4	5
Magnetic Susceptibility $\text{emu}\cdot\text{g}^{-1}$	8.963×10^{-7}	2.961×10^{-5}	2.016×10^{-4}	2.836×10^{-4}	4.241×10^{-4}
Hardness %	97.98	98.90	98.95	99.04	98.97
Moisture %	4.38	4.21	4.24	4.27	4.25
Ash %	1.04	1.36	1.68	4.02	4.89
Iodine value $\text{mg}\cdot\text{g}^{-1}$	1028	1026	1016	968	920
Total iron %	0.15	0.50	0.70	0.85	0.95
Chloride %	0.03	0.06	0.06	0.07	0.06
Acid soluble %	2.47	1.84	2.16	2.92	3.43

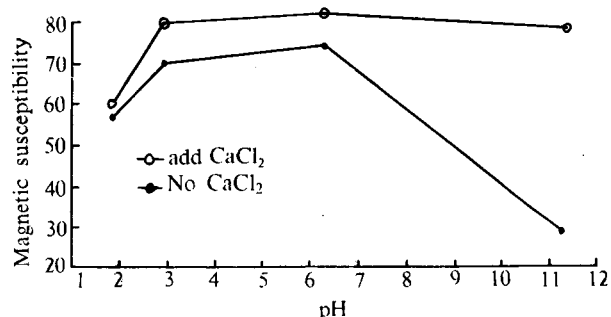


Fig. 3. Calcium chloride for the activation of activated carbon on MR-20 adsorption

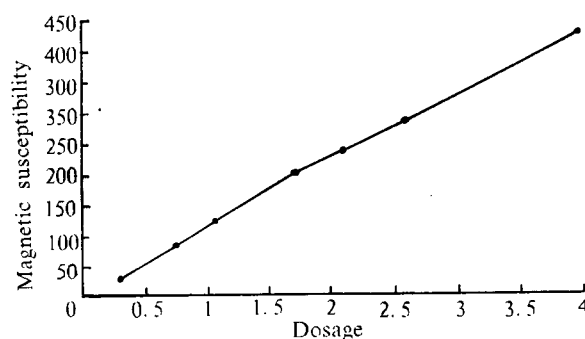


Fig. 4. Magnetic enhancement of activated carbon at various MR-20 dosage

Table 3. Main Indexes of MGH-14

Analytic index	1	2	3	4	5
Magnetic susceptibility $\text{emu}\cdot\text{g}^{-1}$	4.243×10^{-7}	2.638×10^{-5}	2.910×10^{-4}	6.015×10^{-3}	2.978×10^{-1}
Hardness %	97.26	97.31	97.36	97.38	97.20
Moisture %	3.90	3.81	3.84	3.85	3.80
Ash %	0.60	0.92	0.86	1.23	1.36
Methylene blue ml	13.1	13.1	13.0	12.8	12.8
Total iron %	0.02	0.30	0.35	0.45	0.75
Chloride %	0.01	0.04	0.04	0.04	0.04
Acid soluble %	0.47	0.66	1.19	2.11	1.87

Table 4. Main Indexes of MJX-01

Analytic index	1	2	3	4	5
Magnetic susceptibility $\text{emu}\cdot\text{g}^{-1}$	6.071×10^{-7}	0.826×10^{-5}	0.947×10^{-5}	2.588×10^{-5}	3.373×10^{-4}
Hardness %	99.04	99.00	98.90	99.12	98.94
Moisture %	4.29	4.01	4.08	3.97	4.10
Ash %	0.76	1.24	2.59	2.68	3.31
Iodine value $\text{mg}\cdot\text{g}^{-1}$	1045	1041	1032	1027	1004.5
Total iron %	0.10	0.60	0.65	0.80	0.85
Chloride %	0.02	0.05	0.05	0.06	0.05
Acid soluble %	2.47	0.86	1.42	1.64	2.79

References

1. 王崇琳. 发明专利申请公开说明书. CN102302A
2. 公开特字公报. 特开平. 5-200224
3. 磁性活性炭及其在炭浆溢提金工业中的应用. 中国科学院金属研究所. 2309 课题组
4. 王会宗等. 1989. 磁性材料及其应用. 国防工业出版社.
5. Hwang, J. Y. 1992. Magnetic Reagent for Mineral Processing. For presentation at the SME Annual Meeting phoenix Arizona February. 24. 27.
6. Berkowite, A. E. et al. 1980. IEEE TRans. Magn. Vol. MAG-16 No. 2. . 184
7. S. E knalala G. W. Remiers. U. S. P. 3764540. 1973
8. R. E. Rasenweig U. S. P. 3917538
9. Elmre. W. C. 1938. Physical Review Series*11. vol. 54 P309.
10. Whitehead U. S. P 4554088. 1985
11. J. Y. Hwang U. S. P 4834898. 1989
12. J. Y. Hwang U. S. P 4906382. 1990

(Responsible Editor: Dai Fangtian)